

ORGANIC ANALYSIS.

Analysis of Commercial Calcium Citrate. I. Barboni. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 311-341.)—From the results of a critical study of the various methods proposed for the examination of commercial calcium citrate, the conclusion is drawn that the method of Spica (*ANALYST*, 1910, **35**, 519) is the most accurate and rapid. The method of Patané, which is similar to that of Gadais (*ANALYST*, 1909, **34**, 214), is accurate in the absence of tartrates, for which a preliminary qualitative test should be made. The modification of Gadais has the advantage of effecting complete precipitation by the addition of alcohol in such proportion that eventually a liquid is obtained containing 45 per cent. of alcohol, in which the citrate is insoluble.

C. A. M.

Valuation of Fusel Oil. G. Heinzelmann. (*Zeitsch. Spiritusind.*, 1912, 35, 612-613; through *J. Soc. Chem. Ind.*, 1912, 31, 1142.)—Raw fusel oil should be

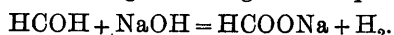


bought on the basis of its content of amyl alcohols (b.-pt. 127° to 131° C.) which varies greatly in commercial samples. The author describes a method for determining the content of amyl alcohols by fractional distillation, used in the Institut für Gärungsgewerbe, Berlin. The best form of fractionating column for the purpose is that shown in the figure. The vapour rises in the enlarged portion of the column, outside the inner bulb-tube, and passes into the right-hand bulb, and thence through the spiral tube into the left-hand limb. The portion condensed in the latter limb runs down inside the bulb-tube, and forms a trap in the lower U-shaped portion, which should be at least 12 to 13 cms. long. When used with liquids which boil above 110° C., the column may be surrounded with cotton wool as far up as the spiral tube. The left-hand thermometer usually indicates 1.5° to 2° C. lower than the right-hand one. The method of fractionation employed is illustrated by the following example: 1,500 c.c. of raw fusel oil were distilled and collected in four fractions—viz., 540 c.c. passing over below 90° C., 420 c.c. between 90° and 115° C., 45 c.c. between 115° and 127° C., and 475 c.c. between 127° and 131° C. The first fraction, consisting chiefly of ethyl alcohol, but containing some water and fusel oil, was distilled from a water-bath (with the fractionating column) so long as ethyl alcohol passed over; the residue, which separated into two layers, one aqueous and the other fusel oil, was treated with sodium chloride (about 20 per cent. of the aqueous

portion), and the fusel oil was isolated by means of a separating funnel. The second fraction, which consisted of two immiscible layers, aqueous and oily, was treated in the same way. The fusel oil thus obtained from the first two fractions was mixed and refractionated. When the thermometer indicated 112° C., the third fraction was added and distillation continued. The portion passing over above 127° C. amounted to 175 c.c., which, together with the fourth fraction from the original distillation (475 c.c.), gave a total yield of 650 c.c. of amyl alcohols, corresponding to 43.3 per cent. of the volume of the raw fusel oil. Particulars respecting three other samples of fusel oil are also given.

Interaction between Formaldehyde and Bismuth or Copper Compounds in the Presence of Alkali. F. Hartwagner. (*Zeitsch. anal. Chem.*, 1913, 52, 17-20.)—When formaldehyde is added to bismuth hydroxide, reduction does not take place even when the mixture is heated; if, however, an excess of sodium hydroxide is also added, the whole of the bismuth is precipitated as metal. In the case of bismuth

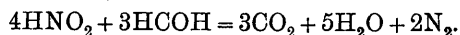
chloride, a large excess of both formaldehyde and sodium hydroxide must be added to insure the complete reduction of the bismuth. Copper may also be precipitated completely in the form of metal from its salts by the combined action of formaldehyde and alkali; it was observed that the finely-divided copper acted as a catalyst, and caused hydrogen to be evolved from the mixture of formaldehyde and alkali, these two substances reacting according to the equation:



Further experiments showed that acetaldehyde in alkaline solution reduces gold and silver salts quantitatively; it also reduces gold salts in acid solution. Chloral hydrate reduces gold, silver, and copper salts completely in alkaline solution, but bismuth salts only partially.

W. P. S.

Interaction between Formaldehyde and Nitrous Acid. L. Vanino and A. Schinner. (*Zeitsch. anal. Chem.*, 1913, **52**, 21-28.)—When an excess of formaldehyde is added to a nitrite solution and the mixture is acidified, the nitrite is decomposed according to the equation:



This reaction forms a means for the estimation of nitrous acid. The nitrite solution is placed in a flask, a delivery-tube from which dips under the lower end of a measuring burette filled with potassium hydroxide solution. The air is expelled from the flask by boiling the solution; the flame is then removed, and clips on the delivery and inlet tubes of the flask are closed. Formaldehyde and hydrochloric acid are now drawn into the flask and the nitrogen evolved is collected in the burette, the last portion of the gas being expelled from the flask by boiling the contents until only steam and hydrochloric acid pass from the delivery-tube. The volume of the gas is then measured with the usual precautions, and its volume calculated into nitrous acid. This method cannot be employed, conversely, for the estimation of formaldehyde, as the reaction only takes place in the presence of an excess of the aldehyde.

W. P. S.

Studies on the Chemistry of Gelatin and its Estimation. M. Berrár. (*Biochem. Zeitsch.*, 1912, **47**, 189-214.)—Water at a temperature of 21° C. dissolves 0.62 per cent. of gelatin; at 18° C., 0.60 per cent.; and at 17° C., 0.56 per cent.; the quantity of gelatin which is soluble in cold water is therefore approximately equal to the smallest amount of gelatin which is necessary to form a jelly. If pieces of gelatine be added to a saturated gelatin solution and the temperature is lowered, a portion of the dissolved gelatin separates out and no jelly is formed. Gelatin is not precipitated completely by alcohol or metaphosphoric acid, and an excess of the latter dissolves the precipitate first formed. The precipitate obtained when albumin is treated with metaphosphoric acid dissolves partially in an excess of the reagent. By precipitation with picric acid and potassium mercuric iodide, the authors find that the molecular weight of gelatin is 823. Gelatin is precipitated quantitatively from its aqueous solution by the addition of an equal volume of saturated picric acid solution, provided that the temperature

of the mixture is below 8°C .; at higher temperatures even a large excess of picric acid does not precipitate the whole of the protein. The nitrogen in the precipitate may be estimated by Kjeldahl's method after reduction with iron turnings and acetic acid. The gelatin-picric acid compound dissolves in a 2 per cent. urea solution containing sodium chloride, and picric acid is therefore useless for the precipitation of gelatin in urine. Alcohol also dissolves the compound readily, and this forms a means of separating gelatin from albumin, albumoses, peptones, mucin, and casein, as the picric acid compounds of these substances are insoluble. For the estimation of gelatin in admixture with other proteins, it is recommended that an aqueous solution of the mixture be treated with a solution consisting of 1 part of saturated picric acid solution and 4 parts of alcohol; albumin, albumoses, peptones, mucin, and casein, are precipitated, and, after they have been removed by filtration, the gelatin is precipitated in the filtrate by cooling the latter below 8°C . and adding an excess of picric acid. This method may also be used for the detection of traces of gelatin in the presence of other proteins, as 1 part of gelatin per 100,000 parts of solution will yield a distinct turbidity on the addition of an excess of picric acid.

W. P. S.

Influence of Antiseptic Agents on the Reichert-Meissl Value of Fats. C. Grimaldi. (*Ann. Lab. Chim. Centr. delle Gabelle*, 1912, **6**, 631-640).—An addition of benzoic acid to butter-fat, oleomargarine or cocoanut oil raises their Reichert-Meissl values, the increase being proportional to the amount added. In the usual proportions (0.1 to 0.2 per cent.) found in edible fats the influence of benzoic acid is insignificant. Salicylic acid also increases the Reichert-Meissl value of oleomargarine and cocoanut oil, but lowers the value of butter-fat when present in greater quantity than 0.5 per cent. In the usual proportion of about 0.1 per cent., salicylic acid has little appreciable influence upon the Reichert-Meissl value of any of the three fats. Attention is called to the fact that by the use of benzoic acid it is possible to prepare a mixture of fats giving the analytical values of genuine butter-fat. For example, a mixture of 78 per cent. of butter-fat (Reichert-Meissl value = 27.4), 9 per cent. of oleomargarine, 9 per cent. of cocoanut oil, and 4 per cent. of benzoic acid, gave the following results: Reichert-Meissl value, 26.1; saponification value, 230.7; refractive index at 40°C . (Zeiss), 44.1; and Maumené figure, 30.8.

C. A. M.

Modification of Halphen's Test for Cottonseed Oil. E. Gastaldi. (*Ann. Lab. Chim. Centr. delle Gabelle*, 1912, **6**, 601-609).—By substituting a small quantity of pyridin for the amyl alcohol in Halphen's reagent the test is rendered much more sensitive and the colorations more intense. A drop of pyridin is added to 5 c.c. of the oil under examination, and then 4 c.c. of carbon bisulphide containing 1 per cent. of sulphur in solution, and the tube heated in the water-bath for fifteen to thirty minutes. With an oil containing as little as 0.25 per cent. of cottonseed oil a yellowish-rose coloration is obtained, whilst Halphen's original reagent only gives a yellowish colour under the same conditions. The reaction is rendered more intense by heating the mixture in a closed vessel. As in the case of the original test, no

reaction is obtained with cottonseed oil that has been heated to 250° C., or has had a prolonged treatment with sulphur dioxide. C. A. M.

Oxidation Assay of Essential Oils. F. D. Dodge. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 6, 86-92.)—It is possible by selective oxidation with potassium permanganate solution to estimate the constituents in certain essential oils. For example, on chilling 10 c.c. of an oil containing eucalyptol (cineol) in a litre flask with a narrow neck, and adding saturated potassium permanganate solution in small portions (25 c.c.), so long as reduction takes place the eucalyptol is unaffected, whereas the other constituents of the oil are oxidised to soluble products. During the addition the flask is continually shaken and kept cool by means of ice, and when the liquid is no longer decolorised after standing for a few hours, the precipitated manganese oxide is dissolved by the addition of sulphurous acid solution (or sodium bisulphite and hydrochloric acid), care being taken to avoid an excess. The flask is then filled with water so as to bring the unoxidised oil into the neck, and this oil finally transferred by means of a capillary pipette into a graduated measuring-tube. In this way the eucalyptol in eucalyptus and cajuput oils may be quantitatively estimated. Rosemary oil yielded about 35 per cent. of unoxidised oil, containing, in addition to eucalyptol, camphor and bornyl acetate, which can be estimated in the usual manner. Spike (lavender) oil gave 41 per cent. of unoxidised oil consisting of eucalyptol and camphor. Test estimations with mixtures containing eucalyptol and terpenes showed that the latter were completely removed in the process. The fenchol in fennel oil is also unoxidised, but the separation is difficult owing to the formation of anisic acid (from the anethol) which separates as a crystalline deposit or is converted into manganese anisate. In the case of oils containing borneol or its esters, the latter are usually stable, whilst the free borneol is converted into camphor. The following amounts of unoxidised oil were separated: Pine needle (Siberian), 60; spruce, 45; sage, 16; and valerian (Japanese), 62 per cent. Menthol is readily oxidised to menthone, which is then slowly but completely oxidised. Menthyl acetate, however, is not attacked. All terpenes, with the exception of camphene, are rapidly oxidised. The slow rate at which camphene is oxidised enables it to be separated in this way in certain cases. Of other oils, cardamom left 8 per cent. (principally eucalyptol); pennyroyal, 5 to 10 per cent. (apparently menthone or a similar ketone); rue, 90 per cent.; cedar, 40 per cent.; copaiba (Para), 24 per cent. of unoxidised oil; and sandal (East Indian), a mixture of oil and a crystalline acid. Cymene is very slowly oxidised, and may be separated to a large extent from terpenes. The aldehydes of bitter almond and cassia oils are completely oxidised to benzoic acid, whilst cumin yields about 20 per cent. of cymene with terephthalic acid. In the case of phenols some yield simple oxidation compounds, whilst others form intermediate derivatives. Sassafras oil yields 5 per cent. of unoxidised oil containing camphor, and parsley and calamus oils from 5 to 6 per cent.

In applying the method to the detection of petroleum products in oil of turpentine, it was found that 10 c.c. of the latter require from 800 to 850 c.c. of the permanganate solution for oxidation. It is also possible to detect petroleum products

in citronella oil in this way. The unoxidised part of the oil (which may normally contain camphor derived from the borneol) is treated with cold 80 per cent. sulphuric acid to eliminate the camphor, and the insoluble paraffins recognised by the fact of their not mixing with castor oil at 0° C. A further use of the process is to distinguish between bornyl acetate, which is not oxidised, and the isomeric linalyl, geranyl, and terpinyl acetates, which are readily oxidised. By estimating the actual amount of ester in an oil and that left after oxidation, the real amount of bornyl acetate—*e.g.*, in rosemary oil—is obtained. Fenchyl acetate, if present, would probably behave like bornyl acetate. C. A. M.

Studies on Fish Oils. Properties of Fish and Vegetable Oil Mixtures. G. F. White and A. Thomas. (*J. Ind. Eng. Chem.*, 1912, 4, 878.)—The viscosity, fluidity, and density, through a range of temperatures, and the refractive index, and acid, saponification, and iodine (Hübl) numbers, of dogfish liver, soya bean, linseed, and China wood oils, and mixtures of these, were determined. The fluidities (the reciprocals of the viscosities) of fish and vegetable oil mixtures were found to be additive, except when either component decomposes on heating, as in the case of China wood oil; the viscosities are also additive if the viscosities of the components approximate each other closely. As has been shown already for fish oils, the fluidities are nearly proportional to the temperature. The fluidity of China wood oil is low, and is a criterion of its purity, as even small quantities of the other oils would raise its fluidity to an extent readily detected. The densities of the mixtures are approximately additive, and are directly proportional to the temperatures; China wood oil has a very high density. No general conclusions can be drawn from the refractive index, acid number, and saponification number. Linseed oil has a higher acid number than dogfish oil, and China wood oil a lower one. Small quantities of fish oil lower the refractive index of China wood oil. The iodine value is lowered by the addition of dogfish liver oil to vegetable oils. O. E. M.

Detection of Petroleum Products in Oil of Turpentine by Means of the Maumené Test. R. Massy. (*J. Pharm. Chim.*, 1912, 6, 484-491.)—In Tortelli's method of testing oil of turpentine, 10 c.c. of the sample are treated in a Dewar's vacuum-tube with 10 c.c. of sulphuric acid (sp. gr. 1.722), the mixture stirred with a thermometer, and a note taken of the rise in temperature. Tortelli found that genuine commercial samples gave figures ranging from 100° to 105° C., and that in this way it was possible, not only to detect the presence of adulterants, but also to ascertain whether a pure oil had been altered by exposure to light and air. Oxidised oils gave a higher figure, the temperature rising more suddenly, whilst the residual mass in the tube was black and viscous instead of being only of a dark colour and fluid. In the author's experiments with oil of turpentine from *Pinus pinaster* (the only kind official in France), the Maumené figure with a pure oil was 96.5° C., whilst an addition of 10 per cent. of various petroleum products lowered the figure considerably. When other pure oils from *P. pinaster* were tested, however, results ranging from 93.2° to 106.0° C. were obtained. It would thus be possible to prepare mixtures of certain

turpentine oils with 10 per cent. of white spirit giving figures well within these limits. To make the test reliable, the sample should be distilled so as to eliminate the bulk of the constituents other than pinenes, and the first 80 per cent. of the distillate collected. In the case of the oil from *P. pinaster* this distillate will consist of a mixture of α - and β -pinene in variable proportions, and will give fairly constant Maumené figures. Thus, fifteen samples gave results ranging from 96.2° to 100.6° C. A pure sample giving a value of 98.8° C. was mixed with 10 per cent. of various adulterants, and the distillate tested, with the following results: Turpentine oil with lamp oil, 91.3° C.; with rectified petroleum spirit, 84.6° C.; with ligroin, 82.2° C.; and with white spirit, 88.3° C. As little as 4 per cent. of white spirit was invariably detected in the test experiments.

C. A. M.

Estimation of Sucrose in the Presence of Other Sugars. G. Testoni. (*Ann. Lab. Chim. Centr. delle Gabelle*, 1912, **6**, 582-589.)—The method devised by Margaillan (*ANALYST*, 1910, **35**, 315) gives good results with mixtures of pure lactose with other sugars, optically inactive lactic acid being formed, but is not satisfactory when applied to natural products, such as milk. When the Bulgarian bacillus is cultivated in milk, a mixture of dextro-rotatory and lævo-rotatory lactic acid is produced, in which the former predominates. The effect of this may be eliminated by removing the lactic acid by means of zinc sulphate. For example, in the case of condensed milk 20 grms. are dissolved in hot water, and the solution treated with acetic acid to coagulate proteins, then with basic lead acetate, followed by an excess of a saturated solution of sodium sulphate, and made up to 202 c.c., an allowance of 2 c.c. being made for the bulk of the coagulated compounds. The liquid is then filtered, and 100 c.c. of the filtrate treated with malto-peptone and 3 grms. of precipitated chalk, and the mixture neutralised, sterilised, infected with the Bulgarian bacillus, and incubated for six days at 35° C. Then, after concentration and cooling, the lactic acid is precipitated by the addition of a saturated solution of zinc sulphate, and the liquid made up to 100 c.c. and rapidly filtered. The sucrose may then be estimated polarimetrically in the filtrate. In a test experiment with a sample of condensed milk, the amount of sucrose thus found was 34.70 per cent. as against 34.84 per cent. found by Clerget's method. The alkali method of Jolles (*ANALYST*, 1911, **36**, 28) is shown to give good results with condensed milk.

C. A. M.

Method for the Direct Polarimetric Determination of Sucrose in Presence of Certain Reducing Sugars. P. Lemeland. (*Zeitsch. Ver. deut. Zuckerind.*, 1912, 1192-1196; through *J. Soc. Chem. Ind.*, 1912, **31**, 1046.)—The optical rotatory and cupric-reducing powers of alkaline solutions of arabinose, dextrose, lævulose, galactose, and lactose are destroyed completely by heating with hydrogen peroxide in presence of a little manganese dioxide. Sucrose and dextrans are not affected by this treatment. For the determination of sucrose in presence of any of the reducing sugars mentioned above, a solution of the mixture is heated in a boiling water-bath with 0.5 gm. of manganese dioxide, or with 2 grms. if the quantity of reducing sugar present exceeds 10 grms. For every gm. of reducing sugars present, a mixture of 2 c.c. of 36 per cent. sodium hydroxide solution and

30 c.c. of hydrogen peroxide (German Official preparation) is added little by little to the hot liquid; this operation may be completed in thirty-five to forty-five minutes. The solution is then cooled, neutralised exactly with dilute acetic acid, decolorised with charcoal or basic lead acetate, made up to a convenient volume and polarised. The author cites a case in which a solution of 2 grms. of sucrose and 20 grms. of dextrose in 100 c.c. was treated with 2 grms. of manganese dioxide, 20 c.c. of sodium hydroxide, and 600 c.c. of hydrogen peroxide, as described. After cooling, nearly neutralising, concentrating to 400 c.c. on a water-bath, decolorising, and filtering, a polarimetric reading of $+42'$ was obtained instead of $+40'$ (control solution of sucrose alone). In analysing mixtures containing a high proportion of reducing sugars to sucrose, advantage may be taken of the solubility of sucrose in 91 per cent. alcohol, as in the following example: A mixture of 1 gm. of sucrose with 25 grms. of dextrose was left for twenty-four hours in contact with 100 c.c. of 91 per cent. alcohol and shaken frequently. The undissolved portion was separated by centrifuging, and washed twice with 50 c.c. of 91 per cent. alcohol. The combined alcoholic solutions were distilled, after addition of a trace of calcium carbonate, and the residual liquid was treated with the oxidising mixture as prescribed above. A polarimetric reading of $+1^{\circ} 14'$ was obtained instead of $+1^{\circ} 18'$ (control solution of pure sucrose). The new method cannot be applied to mixtures containing maltose, for this sugar is not completely decomposed by the oxidising mixture. Dextrins can be determined in presence of sugars other than maltose, any sucrose present being first inverted.

New Technical Determination of Bleaching Quality of Sulphite Pulp.

E. Richter. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 12, 233-236.)—The author had previously standardised Hempel's method for the estimation of lignin in cellulose pulps by heating with 13 per cent. nitric acid and measuring the gases produced, or titrating the nitrous acid with permanganate. He now describes a colorimetric method based on the same reaction, after having proved that the coloration produced is, under prescribed conditions, proportional to the percentage of residual lignin, and that this, again, is proportional to the consumption of bleaching-powder by the sulphite pulp under examination. Five grms. of the air-dry pulp are disintegrated, placed in a wide-necked bottle with 100 c.c. of 13 per cent. nitric acid, thoroughly shaken, and kept for about an hour in a dark place. After that time the bottle is again shaken, and the pulp is filtered off on a dry funnel with a plug of cotton-wool. Twenty-five c.c. of the filtrate are placed in a small bottle. A standard pulp of similar grade and of known bleach consumption is treated in the same manner. Water is added from a burette to the darker liquid until both solutions are identical in colour. Suitable amounts of each liquid are compared in Eggertz carbon tubes, looking down from above against white paper placed below, and using different amounts—5 c.c. and 10 c.c. Should the colours not be uniform, the shades may be adjusted by varying the depth of the column of liquid in one of the tubes. The bleach consumption of the test sample is then readily calculated by simple proportion compared with the standard sample. A very highly purified pulp may not produce sufficient coloration after one hour at the laboratory temperature; in

such a case it is permissible to place the bottles in a water-bath at 40° C., at which temperature a coloration is developed in about fifteen minutes. J. F. B.

Estimation of the "Degree of Bleaching" [Chemical Condition] of Cotton. C. Piest. (*Zeitsch. angew. Chem.*, 1912, **25**, 2518-2522.)—In the nitro-cellulose and artificial silk industries it is necessary to know the degree of chemical modification of the bleached cotton used as raw material. A number of empirical methods of analysis have been standardised by various authors, the majority of which are described in detail in Schwalbe's "Die Chemie der Cellulose." The author has carried out determinations of the more important of these "constants," first upon a series of cottons modified in known directions, and secondly upon a series of commercial bleached cotton wastes submitted for nitration purposes. The principal "constants" studied were—(1) The wood-gum value—*i.e.*, the constituents soluble in cold 5 per cent. sodium hydroxide, and reprecipitated on acidification and addition of alcohol; (2) Schwalbe's total copper value (boiling Fehling's solution); (3) the cupric hydroxide value—*i.e.*, the amount of copper absorbed from cold Fehling's solution; (4) Vieweg's acid value—*i.e.*, the amount of sodium hydroxide neutralised on boiling for fifteen minutes with a 1 per cent. solution; (5) the amount of cupric hydroxide absorbed from a 10 per cent. solution of copper sulphate; (6) Ost's viscosity value, representing the viscosity of solutions of the cotton in cuprammonium solution prepared under rigidly prescribed conditions. The results obtained with cottons specially modified are summarised in the following table:

Nature of Cotton.	Schwalbe's Copper Value.	Cupric Hydroxide Value.	Vieweg's Acid Value.	Copper Sulphate Value.	Ost's Viscosity Test. Solution diluted—	
					After 24 Hours.	After 48 Hours.
1. Normally prepared ni- trating cotton	{ 1.64 1.64	{ 0.53 0.57	{ 1.86 1.86	{ 2.58	{ 26.50 24.40 22.00	{ 17.35 16.50 15.77
2. Overbleached cotton	{ 8.76 8.76	{ 0.25 0.25	{ 6.48 6.40	{ 1.54	{ 2.70 2.70 2.70	{ 2.58 2.58 2.58
3. Very strongly oxidised cotton	{ 16.50 16.00	{ 0.52	{ 9.22	{ 1.08	{ 2.23 2.23 2.23	{ 2.12 2.12 2.12
4. Sample No. 2 digested for twenty-four hours with cold 5 per cent. sodium hydroxide	{ 3.79 3.79	{ 0.17 0.17	{ 4.30 3.65 3.86	{ 2.97	{ 2.64 2.64 2.64	{ 2.58 2.58 2.58
5. Mercerised cotton	{ 1.52 1.52	{ 1.47 1.47	{ 0.40	{ 1.87 1.91	{ 17.46 16.15 14.62	{ 14.15 13.45 12.88
6. Hydrocellulose prepared with sulphuric acid, sp. gr. 1.53 in the cold	{ 4.13 4.13	{ traces	{ 7.04 7.04	{ 2.33 2.33	{ 2.65 2.65 2.65	{ 2.43 2.43 2.43

The above figures, in conjunction with others obtained with commercial samples of normally bleached nitrating cottons, lead to the following conclusions: Normally bleached cotton should show a "wood-gum" value not exceeding 1; the total copper value should not exceed about 1.5, and preferably should not be higher than 1; from this is to be deducted the cupric hydroxide value, which, except for mercerised cotton, should range round about 0.5. Vieweg's acid value, which is a measure of the constituents readily susceptible to hydrolysis by alkalis, should be considerably less than 2, preferably less than 1.5. Ost's viscosity value is an extremely delicate test for chemical condition; this value decreases the longer the solution is allowed to stand before dilution prior to making the observation. Moreover, in the case of normal cottons, when three estimations are made consecutively, each observation is lower than the previous one. The author considers that normal cottons should show a viscosity of at least 10 when the solution is allowed to remain for five days before dilution; this period is selected because the differences between three consecutive observations are then less pronounced. Modification by overbleaching (oxidation) is shown by an abnormally high copper value, a low or normal hydroxide value, a high acid value according to Vieweg, and a low viscosity. Extraction of the constituents of the oxycellulose soluble in alkali lowers the copper value, which, however, still remains abnormal. Vieweg's acid value is changed in a similar manner, but the viscosity value is unchanged. Mercerisation increases the cupric hydroxide value, which is a specific criterion of hydrated cellulose; Vieweg's acid value is lowered owing to the elimination of hydrolysable constituents by the strong mercerising lye, whilst the viscosity value is not appreciably lowered by the short action of mercerising alkali. Modification by the action of acids (hydrocellulose) results in an increase of the copper value, but in less degree than modification by oxidising agents; the cupric hydroxide value is lowered, but the sensitiveness to alkaline hydrolysis (acid value) is very high in relation to the copper value; the viscosity is lowered, just as with overbleached cellulose. The author considers that the copper sulphate value is of subordinate importance as a diagnostic test.

J. F. B.

Researches on the Gelatinising Temperatures of Starch Granules.
M. Nyman. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 673-676.)—In a first series of experiments various starches were mixed with water heated to a suitable temperature, and as the temperature was raised drops of the mixtures were removed every thirty seconds and examined under the micro-polariscope. The point at which the granules ceased to show interference crosses was taken as the gelatinising temperature. Rye, barley, and wheat starches were found to gelatinise at slightly different temperatures, the results obtained being—Rye starch, 57° C.; barley starch, 58° C.; and wheat starch, 59° C. Further experiments on the rate at which the gelatinisation proceeded showed that the larger starch granules gelatinised more quickly than the smaller. Although the differences in the gelatinising temperatures does not afford a means of identifying two starches in a mixture of the same, the rate of gelatinisation may be of some use for the purpose. If a mixture of rye and wheat meals is maintained at a temperature of 53° C., the rye starch is gelatinised in six minutes, whilst the

wheat starch requires twenty-four minutes ; any ungelatinised starch granules observable in such a mixture after seven minutes' heating indicate the probable presence of wheatmeal.

W. P. S.